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RESEARCH ARTICLE

**ZHANG Baoli, XING Yongheng, ZHANG Yuanhong, BAI Fengying, XU Jiqing, YANG Guangdi, HUANG Xiaoying**

# Syntheses, spectra and crystal structures of a series of Mo/S(O) complexes containing oxalate ligand

**Abstract** Two Mo/S complexes: $K_6[Mo_2O_2S_2(C_2O_4)_2(ClPO_3)_2]\cdot H_2O$ (**1**) and $K_6[Mo_2O_3S(C_2O_4)_2(ClPO_3)_2]\cdot 3H_2O$ (**2**) containing oxalate ligand were prepared by the reaction of $(NH_4)_2MoS_4$ and potassium oxalate by adding a small amount of $H_3PO_4$ to adjust the value of pH in the system of methanol and water under an atmosphere of pure nitrogen at ambient temperature and characterized by means of elemental analysis, IR, and UV–Vis. Results of the analysis reveal that crystals of the complex 1 are orthorhombic, space group *Pbcn*, $a = 4.6370(2)$ nm, $b = 0.7549(2)$ nm, $c = 1.3888(4)$ nm, $V = 4.861(2)$ nm$^3$, $Z = 8$, $M = 945.49$, $Dc = 2.58$ g/cm$^3$, $\mu = 2.625$ cm$^{-1}$, $R = 0.063$, and $wR = 0.075$. In addition, their spectral properties were also discussed.

**Keywords** oxalate ligand, Mo/S(O) complex, spectral properties, crystal structure

## 1 Introduction

Oxalic acid, an important organic ligand, tends to form many metal complexes with a versatile molecular structure [1–3]. The study of Mo complexes with oxalate as ligand has been attracting interest, the reasons being as follows: (i) the structure of Mo complexes with oxalate is versatile. Even if they have the same composition, their structures are different. A compound of $K_2[MoO_3(C_2O_4)]\cdot H_2O$ [4] was reported and its structure has two types: discrete cluster structure and infinite chain structure [5,6]. (ii) Mo complexes with oxalate have the characteristic property of luminescence induced by chemistry or electron [7–9], Mentzen et al. have studied widely on this aspect [4]. (iii) Mo complexes with oxalate are related to the catalysis reaction of nitrogen-fixing enzymes; on the other hand, it is related to life chemistry. It can deoxidize $N_2H_4$ to $NH_4^+$. In 1994, Demadis and Coucouvanis [10,11] studied and explored in this area, which attracted more interest on these type of complexes. To our knowledge, the report on Mo/S complexes containing oxalate ligand is rare, especially for Mo or W poly(carboxyl) ligand complexes containing S-bridge. Our group has done a great deal of work on the study of Mo or W complexes containing poly(carboxyl) ligand, including the synthesis, crystal structure, and spectrum character of a series of Mo or W complexes [12–14]. In this paper, two new complexes, $K_6[Mo_2O_2S_2(C_2O_4)_2(ClPO_3)_2]\cdot H_2O$ and $K_6[Mo_2O_3S(C_2O_4)_2(ClPO_3)_2]\cdot 3H_2O$, were synthesized, which may help to know the structure and function of Mo or W complexes containing oxalate.

## 2 Experimental

### 2.1 General procedure

The content of K and Mo were performed on AA-457 atomic absorption spectrum, the content of P and S were performed on Plasma-Spec(I)-AES ICP analyzer. The content of chlorine anion was determined by chemical volume analysis. Elemental analyses (C, H, and N) were performed on a PE 240C analyzer. Infrared spectra were recorded on an Elmer783 spectrometer in the spectral range 4000–220 cm$^{-1}$ with the samples in the form of potassium bromide pellets. The UV–Vis spectra were recorded on UV-3100 spectrometer in $H_2O$ solution. $(NH_4)_2MoS_4$ was synthesized according to Ref. [15]. All synthetic manipulations were carried out in the atmosphere at room temperature. All solvents were of analytical reagent grade.

### 2.2 Preparation of $K_6[Mo_2O_2S_2(C_2O_4)_2(ClPO_3)_2]\cdot H_2O$ (1)

$K_2C_2O_4\cdot H_2O$ (2.0 g, 10.86 mmol), $H_3PO_4$ (2 mL), $H_2O$ (60.0 mL), and $CH_3OH$ (90.093 mL) were added in a

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ZHANG Baoli, XING Yongheng (✉), ZHANG Yuanhong,
BAI Fengying
College of Chemistry and Chemical Engineering, Liaoning Normal University, Dalian 116029, China

XU Jiqing, YANG Guangdi
College of Chemistry, Jilin University, Changchun 130000, China

HUANG Xiaoying
State Key Laboratory of Structural Chemistry, Fuzhou 350000, China
E-mail:yhxing2000@yahoo.com

rockered flask (250 mL), the pH value of the mixture being 5. After its complete dissolution, $(NH_4)_2MoS_4$ (1.03 g, 4 mmol) and KCl (2.0 g, 26.8 mmol) were added to the above solution. The mixture was stirred for about 48 h at room temperature, under an atmosphere of nitrogen, the color of the resulting solution was changed into dark red, and black precipitates were produced. Finally, the black precipitates were filtered off (black precipitates could be a small quantity of raw material of $(NH_4)_2MoS_4$ and simple Mo/S complex, which were confirmed on the data of IR spectra), a red solution was discovered, the pH value was about 7, airproofed from air and protected with nitrogen gas, and stood at room temperature for several days (during this period, filted off one or two times). Dark red petal crystals were grown, which can be affirmed primarily as the title complex **1** accoording to the data of IR spectra. The molecular formula of complex **1** could be suggested as $Mo_2K_6Cl_2S_2P_2O_{17}C_4H_2$. Anal. Calcd. for $Mo_2K_6Cl_2S_2P_2O_{17}C_4H_2$: Mo, 20.00; S, 6.60; K, 25.01; P, 6.59; Cl, 7.52; C, 5.05; H, 0.24. Found: Mo, 20.29; S, 6.77; K, 24.75; P, 6.56; Cl, 7.50; C, 5.08; H, 0.21. IR (KBr, $cm^{-1}$): $v_{\mathrm{OH}}$, 3585; $v_{\mathrm{asC=O}}$, 1680, 1665, 1651; $v_{\mathrm{sC=O}}$, 1435, 1409; $v_{\mathrm{Mo=O}}$, 931, 909, 903; $v_{\mathrm{P-O}}$, 1185, 1171, 1141; $v_{\mathrm{Mo-Sb}}$, 459.

### 2.3 Preparation of $K_6[Mo_2O_3S(C_2O_4)_2(ClPO_3)_2]\cdot 3H_2O$ (**2**)

$K_2C_2O_4\cdot H_2O$ (0.7 g, 3.8 mmol), $H_3PO_4$ (2 mL), $H_2O$ (50.0 mL), and $CH_3OH$ (50.0 mL) were added in a rockered flask (250 mL), the pH value of the mixture was adjusted to 7.5 with 1 mol/L KOH. $(NH_4)_2MoS_4$ (1.03 g, 4 mmol) and KCl (2.0 g, 26.8 mmol) were added to the above solution. The mixture was stirred for about 48 h at room temperature, under an atmophere of nitrogen, the color of the solution changed into dark red, and black precipitates were produced. Finally, the black precipitates were filtered off (it could be a small quantity of raw material $(NH_4)_2MoS_4$ and simple Mo/S complexes, which were confirmed on the data of IR spectra), a red solution was discovered, the pH value of the solution was about 7.6, airproofed from air and protected with nitrogen gas, stood at room temperature for several days (during this period, filted off for one or two times and 50 mL $CH_3OH$ was added again). Dark red pole crystals were obtained, it can be affirmed primarily as the title complex **2** by the data of IR spectra. The molecular formula of complex **2** could be confirmed as $Mo_2K_6Cl_2SP_2O_{20}C_4H_6$. Anal. Calcd. for $Mo_2K_6Cl_2SP_2O_{20}C_4H_6$: Mo, 20.05; S, 3.40; K, 24.44; P, 6.50; Cl, 7.55; C, 2.35; H, 0.65. Found: Mo, 19.87; S, 3.31; K, 24.24; P, 6.42; Cl, 7.35; C, 2.49; H, 0.62. IR (KBr, $cm^{-1}$): $v_{\mathrm{OH}}$, 3446; $v_{\mathrm{asC=O}}$, 1681, 1666, 1652; $v_{\mathrm{sC=O}}$, 1435, 1409; $v_{\mathrm{Mo=O}}$, 933, 904; $v_{\mathrm{P-O}}$, 1193, 1178, 1156; $v_{\mathrm{Mo-Sb}}$, 461; $v_{\mathrm{Mo-Ob}}$, 788, 709, 494.

### 2.4 X-ray data collection and refinement of the crystal structures for 1 and 2

A crystal of **1** with 0.22 mm × 0.15 mm × 0.15 mm was mounted on a glass fiber for X-ray measurement. Reflection data were collected at room temperature on an Enraf-Nonius $CAD_4$ diffractometer with graphite-monochromated Mo K$\alpha$ radiation ($\lambda = 0.7107$ Å) and an $\omega$-$2\theta$ scan mode. Intensity data were corrected for Lorentz-Polarization effects and absorption ($\varphi$ scans).

The structures were solved by the direct method using SHELXS-86 and refined using SHELXL-97. All the non-hydrogen atoms were refined anisotropically and the contributions of the hydrogen atoms were included in calculated positions, constrained to ride on their carbon atoms with group Uiso values assigned. Atomic scattering factors and anomalous dispersion terms were as in SHELXL-97. The drawings were made with ORTEP32, and all calculations were performed on a pentium4 computer.

Crystallographic parameters for **1**: orthorhombic, space group *Pbcn* ($a = 4.6370(2)$ nm, $b = 0.7549(2)$ nm, $c = 1.3888(4)$ nm) $V = 4.861(2)$ $nm^3$, $Z = 8$, $Dc = 2.58$ $g/cm^3$, $\mu = 2.625$ $cm^{-1}$, $\Delta\rho_{\mathrm{max/min}} = 1.89/-2.11$ e·Å$^{-3}$, $R = 0.063$, and $wR = 0.075$. For complex **2** also, the crystal structure was determined by X-ray diffraction, but the quality of the crystal was not good, the crystal data were not suitable for the request of the publication. While from the rude structure of complex **2**, we can see that it is similar to the structure of complex **1**, only the bridged atom is different. Selected bond distances and angles of complex **1** are listed in Table 1.

**Table 1** Selected bond distances (nm) and angles (°) for **1**

| | | | |
|---|---|---|---|
| Mo1–Mo2 | 0.2806(2) | Mo1–S1 | 0.2313(4) |
| Mo2–S2 | 0.2310(4) | Mo1–O1 | 0.224(1) |
| Mo1–O2 | 0.2174(9) | Mo1–O5 | 0.168(1) |
| Mo2–O10 | 0.170(1) | Mo2–CI2 | 0.2566(4) |
| Mo2–O7 | 0.218(1) | Mo2–O6 | 0.217(1) |
| Mo1–CI1 | 0.2573(4) | P1–O13 | 0.144(1) |
| P1–O12 | 0.145(1) | P1–O11 | 0.150(1) |
| P2–O15 | 0.143(1) | P2–O14 | 0.144(1) |
| P2–O16 | 0.144(1) | O1–C1 | 0.127(2) |
| O2–C2 | 0.127(2) | O3–C1 | 0.120(2) |
| O4–C2 | 0.123(2) | O6-C3 | 0.128(2) |
| O7–C4 | 0.126(2) | O8-C3 | 0.124(2) |
| O9–C4 | 0.123(2) | C1-C2 | 0.155(2) |
| C3–C4 | 0.154(2) | Cl1-P1 | 0.2036(6) |
| Cl2–P2 | 0.2056(5) | | |
| O6–Mo2–O7 | 73.4(4) | O1- Mo1-O2 | 71.4(3) |
| O15–P2–O14 | 111.1(7) | O15–P2–O16 | 111.4(6) |
| O15–P2–Cl2 | 110.4(5) | O14–P2–O16 | 111.8(7) |
| O13–P1–O12 | 110.7(7) | O13–P1–O11 | 112.1(7) |
| O13–P1–Cl1 | 106.0(5) | O12–P1–O11 | 111.7(8) |
| O12–P1–Cl1 | 108.9(5) | O11–P1–Cl1 | 107.2(6) |
| O14–P2–Cl2 | 104.0(5) | O16–P2–Cl1 | 107.8(5) |
| O5–Mo1–O2 | 89.7(4) | O5–Mo1–S2 | 101.0(3) |
| O5–Mo1–S1 | 104.8(4) | O5–Mo1–Cl1 | 91.2(3) |
| O2–Mo1–S2 | 86.9 (3) | O1–Mo1–S1 | 92.1(3) |
| S2–Mo1–S1 | 103.5(2) | S2–Mo1–Cl1 | 166.9(2) |
| O15–P2–O14 | 111.1(7) | O15–P2–O16 | 111.4(6) |
| O15–P2–Cl2 | 110.4(5) | O14–P2–O16 | 111.8(7) |
| O14–P2–Cl2 | 104.0(5) | O16–P2–Cl2 | 107.8(5) |
| C1–O1–Mo1 | 118.4(9) | C2–O2–Mo1 | 122.1(9) |
| C3–O6–Mo2 | 118(1) | C4–O7–Mo2 | 117(1) |
| O1–Mo1–Cl1 | 80.0(3) | | |

## 3 Results and discussion

### 3.1 Syntheses of both complexes

In the process of synthesizing both complexes, a small amount of $H_3PO_4$ was added to adjust the value of pH, it is unexpected that the ligand of $ClPO_3^{2-}$ was found in title complexes. On the basis of experimental results, we conclude that three different cluster core anions may be present in $H_2O/CH_3OH$ solution, $[Mo_2O_2S_2(C_2O_4)_2(ClPO_3)_2]^{6-}$, $[Mo_2O_3S(C_2O_4)_2(ClPO_3)_2]^{6-}$, and $[MoO_3(C_2O_4)]^{2-}$. Their stabilization changes with the value of pH: pH~7.0 → $[Mo_2O_2S_2(C_2O_4)_2(ClPO_3)_2]^{6-}$, pH~7.5 → $[Mo_2O_3S(C_2O_4)_2(ClPO_3)_2]^{6-}$, pH~8.0 → $[MoO_3(C_2O_4)]_n^{2-}$.

The change for the value of pH and the ratio of the starting material may play an imporant role in the formation of the Mo/O complexes containing oxalate ligand. The synthesis and structure of complex **3** have been reported [16]. The reaction processes are depicted in Scheme 1.

### 3.2 Characterization of complexes

#### 3.2.1 IR spectra

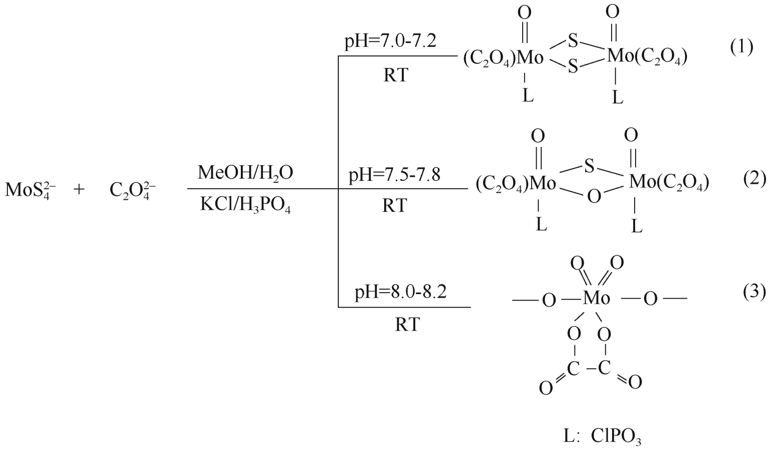
The IR spectra of complexes **1**, **2**, and **3** are listed in Table 2. When compared with free oxalic acid, *cis*- and *trans*-stretching vibrations for C = O of coordinated oxalic acid have an obvious red-shift, showing that the carboxylate group is coordinated to Mo atom. With S atom (coordinated to Mo atom in the form of bridging bonds) substituted gradually by O atom, the frequency of *v*(OH) becomes more and more lower, showing that the extent of combination for $H_2O$ in title complexes increase more and more; the *cis*- and *trans*-stretching vibrations of C = O have a trend to become lower; and the stretching vibration of Mo = O and Mo–Ob have a tendency of intensity degression gradually.

#### 3.2.2 UV–Vis spectra

The UV–Vis spectra of complex **1** is similar to that of complex **2**, having two absorption bands in the range of 190–700 nm. For complex **1**, band at 450 nm ($1.06 \times 10^3$) is attributed to ligand to metal charge transition (S → Mo); similarly, bands at 275 nm ($9.5 \times 10^3$) and 344 nm ($2.7 \times 10^3$) should be assigned ligand to metal charge transition (O → Mo). For complex **2**, band at 450 nm ($2.68 \times 10^2$) is attributed to S → Mo charge transition; bands at 306 nm ($1.86 \times 10^3$) and 350 nm ($1.34 \times 10^3$) are attributed to charge transition (O → Mo) [17]. While for complex **3**, one absorption band was observed at 250 nm, which can be assigned to the charge transfer of O → Mo.

#### 3.2.3 Structural characterization

The crystal structure of complex **1** is shown in Fig. 1. It is composed of six $K^+$, an anion of $[Mo_2O_2S_2(C_2O_4)_2(ClPO_3)_2]^{6-}$, and a lattice molecule of $H_2O$. The anion contains a typical cluster core $[Mo_2O_2S_2]^{2+}$. The bond distance of Mo–Mo is 0.2806(2) nm, the average bond distance of Mo–$S_b$ (b: bridged) is 0.169 nm and the average bond distance of Mo–Ot (t: termal) is 0.169 nm. In addition to the chemical bonds of the cluster core, each Mo atom is coordinated to two oxygen atoms (from a bidentatyl oxalate) and a Cl atom



**Scheme 1** Reaction process of **1**, **2**, and **3**

**Table 2** IR spectra of complexes **1**, **2**, and **3** ($cm^{-1}$)

| | $\nu_{OH}$ | $\nu_{asC=O}$ | $\nu_{sC=O}$ | $\nu_{Mo=O}$ | $\nu_{Mo-Ob}$, | $\nu_{P-O}$ | $\nu_{Mo-Sb}$ |
|---|---|---|---|---|---|---|---|
| 1 | 3585 | 1680, 1665, 1651 | 1435, 1409 | 931 909 903 | | 1185, 1171, 1141 | 459 |
| 2 | 3446 | 1681, 1666, 1652 | 1435, 1409 | 933, 904 | 788, 709, 494 | 1193, 1178, 1156 | 461 |
| 3 | 3490 | 1680, 1643, 1623 | 1432 | 908, 872 | 785, 741, 516, 479 | | |

**Table 3** Comparison of related bond distances (nm) for similar fashion complexes*

| Compounds | Mo–Mo | Mo–Sb (av) | Mo–Ob (av) | Mo–Ot (av) | Mo–O$_{\text{carboxylate}}$ (av) |
|---|---|---|---|---|---|
| **1** | 0.2806(2) | 0.2313(4) | | 0.169(1) | 0.2207(5) |
| **3** | | | 0.1957(2) | 0.1723(3) | 0.2209(2) |
| **4** | 0.2822(1) | 0.2314(2) | | 0.1697(6) | 0.2161(6) |
| **5** | 0.2877(2) | 0.2341(5) | | 0.1707(9) | 0.2200(9) |
| **6** | 0.2823(2) | 0.2328(3) | | 0.1686(8) | 0.2345(7) |

**3**: $K_2[MoO_3(C_2O_4)]\cdot H_2O$ [16]; **4**: $[Mo_2O_2S_2(C_2O_4)_2(H_2O)_2]^{2-}$ [18]; **5**: $K_{2.5}Na_2NH_4[Mo_2O_2S_2(cit)_2]\cdot 5H_2O$ [19]; **6**: $K_2[Mo_2O_2S_2(cys)_2]\cdot CH_3OH\cdot 5H_2O$ [6].

(from $ClPO_3^{2-}$). The bond distances of Mo−O($C_2O_4^{2-}$) are in the range of 0.217(1)−0.224(1) nm, and the bond distances of Mo−Cl are 0.2573(4) and 0.2566(4) nm, respectively. Excluding the bond to Mo–Mo, each Mo atom with two S atoms, three O atoms, and a Cl atom formed a distorted octahedral geometry.

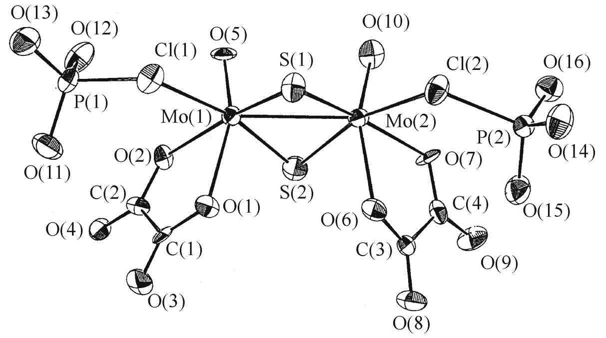


**Fig. 1** Crystal structure of complex **1**, hydrogen atoms, potassium atoms, and lattice waters are omitted for clarity

In the structure of $[Mo_2O_2S_2(C_2O_4)_2(ClPO_3)_2]^{6-}$ P atom, coordinated to one Cl atom and three O atoms, formed a distorted tetrahedral geometry. The distances of P−O are in the range of 0.143(1)−0.150(1) nm, all the bonds of P−O have the characteristics of double bonds. The bond distances of P−Cl are 0.2036(6) and 0.2056(5) nm, respectively.

Complex **2** has a structure similar to that of complex **1**, the only difference being bridge of cluster core. Complex **1** is a Mo/$C_2O_4$ complex containing double sulfur-bridge, while complex **2** is a Mo/$C_2O_4$ complex containing a mixture of one oxygen-bridge and one sulfur-bridge.

Some related bond distances for some Mo complexes are listed in Table 3. The bond distances of Mo−Mo for Mo/S(O) complexes containing oxlate (0.2806(2) nm in complex **1**) are shorter than that of Mo/S(O) complexes containing citrate and cysteine (0.2877(2) nm in complex **5**; 0.2823 (13) nm in complex **6**), and are similar to the distance of Mo−Mo in complex **4**. The distance of Mo−Sb(av) in complex **1** is similar to that in complexes **4** and **6**, but shorter than that in complex **5**. The distances of Mo−Ot(av) in complex **1** are shorter than those in complexes **3** and **5**, similar to those in complexes **4** and **6**. For the distance of Mo−O$_{\text{carboxylate}}$ (av), their length order is $\mathbf{4}<\mathbf{5}<\mathbf{1}<\mathbf{3}<\mathbf{6}$. We can conclude that in Mo complexes with S(O)-bridge, the organic carboxyl group influences the distances of Mo−Ob, Mo−Mo, Mo−Ot, and Mo−O$_{\text{carboxylate,}}$, especially the distance of Mo−Ot.

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ERRATUM

**ZHAO Qiuling, GAO Zhinong**

# Synthesis and surface-active property of bis-quaternary ammonium-sodium sulfate Gemini surfactant

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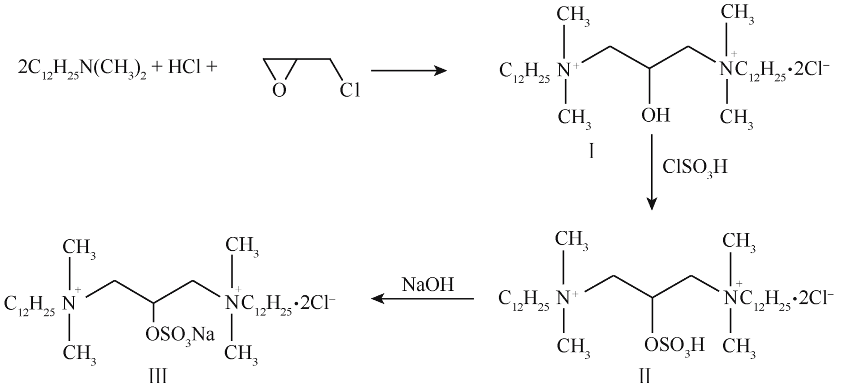
Due to typesettings production errors, some mistakes were made. The Editorial Office regrets these errors.

1) Fig.1 should be changed to as following:



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